Rec

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION
SPONSORED PROJECT INITIATION

Date: 1/4/79

Project Title: High Dose Ion Implantation of Aluminum and Titanium Alloys

Project No: G-41-675 Green Card

Project Director: Dr. J. R. Stevenson

Sponsor: U. S. Air Force Office of Scientific Research (AFOSR); Bolling AFB,D.C. 20332

Agreement Period: From 11/1/78 Until 10/31/79 (Grant Term)

Type Agreement: Grant No. AFOSR-79-0011

Amount: $57,777 AFOSR Funds (G-41-675)
24,862 GIT Contribution (G-41-319)
$82,639 Total

Reports Required: Final Technical Report (Annual Technical if grant extended beyond one year)

Sponsor Contact Person (s):

Technical Matters

Lt. Col. Richard W. Haffner
AFOSR (NC)
Bolling AFB, D.C. 20332

Contractual Matters
(thru OCA)

Capt. David E. McFarlane
AFOSR (PKZ)
Bolling AFB, D. C. 20332

Phone (202) 767-4957

Defense Priority Rating: none

Assigned to: Physics (School/Laboratory)

COPIES TO:

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- Other

CA–3 (3/76)

5K2811

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: 1/27/81

Project Title: High Dose Ion Implantation of Aluminum and Titanium Alloys

Project No: G-41-675

Project Director: Dr. J.R. Stevenson

Sponsor: U.S. Air Force Office of Scientific Research (AFOSR); Bolling AFB, D.C. 20332

Effective Termination Date: 10/31/79 (01 year)

Clearance of Accounting Charges: 10/31/79 (01 year)

Grant/Contract Closeout Actions Remaining:

NONE

_ Final Invoice and Closing Documents
_ Final Fiscal Report
_ Final Report of Inventions
_ Govt. Property Inventory & Related Certificate
_ Classified Material Certificate
_ Other _____

Assigned to: Physics (School/Laboratory)

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Project Code (GTRI)
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Project File (OCA)

CA—4 (1/79)

FINANCIAL STATUS REPORT

(Follow instructions on the back)

1. FEDERAL AGENCY AND ORGANIZATIONAL ELEMENT TO WHICH REPORT IS SUBMITTED	2. FEDERAL GRANT OR OTHER IDENTIFYING NUMBER	OMB Approved No. 80-R0180	PAGE	OF	
Air Force Office of Scientific Research	AFOSR-79-0011		1	1	PAGES

3. RECIPIENT ORGANIZATION (Name and complete address, including ZIP code)	4. EMPLOYER IDENTIFICATION NUMBER	5. RECIPIENT ACCOUNT NUMBER OR IDENTIFYING NUMBER	6. FINAL REPORT	7. BASIS
Georgia Institute of Technology Atlanta, Georgia 30332	58-6002023	G-41-675	☐ YES ☒ NO	☒ CASH ☐ ACCRUAL

8. PROJECT/GRANT PERIOD (See instructions)		9. PERIOD COVERED BY THIS REPORT	
FROM (Month, day, year)	TO (Month, day, year)	FROM (Month, day, year)	TO (Month, day, year)
11/1/78	10/31/79	11/1/78	10/31/79

10. STATUS OF FUNDS

PROGRAMS/FUNCTIONS/ACTIVITIES ►	(a) Personal Services	(b) Retirement	(c) Materials & Supplies	(d) Travel	(e) Capital Outlay	(f) Overhead	TOTAL (g)
a. Net outlays previously reported	$	$	$	$	$	$	$
b. Total outlays this report period	29,980.97	2,253.05	9,298.54	1,511.99	16,500.00	22,785.54	82,330.09
c. *Less:* Program income credits							
d. Net outlays this report period *(Line b minus line c)*	29,980.97	2,253.05	9,298.54	1,511.99	16,500.00	22,785.54	82,330.09
e. Net outlays to date *(Line a plus line d)*	29,980.97	2,253.05	9,298.54	1,511.99	16,500.00	22,785.54	82,330.09
f. *Less:* Non-Federal share of outlays	4,500.13	451.51			16,500.00	3,420.10	24,871.74
g. Total Federal share of outlays *(Line e minus line f)*	25,480.84	1,801.54	9,298.54	1,511.99	-0-	19,365.44	57,458.35
h. Total unliquidated obligations							
i. *Less:* Non-Federal share of unliquidated obligations shown on line h							
j. Federal share of unliquidated obligations							
k. Total Federal share of outlays and unliquidated obligations	25,480.84	1,801.54	9,298.54	1,511.99	-0-	19,365.44	57,458.35
l. Total cumulative amount of Federal funds authorized	25,500.00	1,917.00	9,780.00	1,200.00	-0-	19,380.00	57,777.00
m. Unobligated balance of Federal funds	19.16	115.46	481.46	(311.99)		14.56	318.65

11. INDIRECT EXPENSE	a. TYPE OF RATE *(Place "X" in appropriate box)* ☐ PROVISIONAL ☒ PREDETERMINED ☐ FINAL ☐ FIXED			
	b. RATE	c. BASE	d. TOTAL AMOUNT	e. FEDERAL SHARE
	76%	Salaries & Wages	$22,586.42	$19,365.44

12. REMARKS: *Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation.*

13. CERTIFICATION

I certify to the best of my knowledge and belief that this report is correct and complete and that all outlays and unliquidated obligations are for the purposes set forth in the award documents.

SIGNATURE OF AUTHORIZED CERTIFYING OFFICIAL

DATE REPORT SUBMITTED

TYPED OR PRINTED NAME AND TITLE
David V. Welch, Manager
Grants & Contracts Accounting

TELEPHONE *(Area code, number and extension)*
404/894-4624

269-102

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REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER	2. GOVT ACCESSION NO. / 3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)*: Ion Implantation Studies of Titanium Metal Surfaces	5. TYPE OF REPORT & PERIOD COVERED: INTERIM, Nov. 1, 1978 to Oct. 31, 1979
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): James R. Stevenson, Keith O. Legg	8. CONTRACT OR GRANT NUMBER(s): AFOSR-79-0011
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Georgia Institute of Technology, School of Physics, Atlanta, Georgia 30332	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS: Air Force Office of Scientific Research, Bolling AFB, Bldg. 410, Washington, D. C. 20332	12. REPORT DATE: December 17, 1979
	13. NUMBER OF PAGES: 8
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*	15. SECURITY CLASS. *(of this report)*: Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release:

Distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

TITANIUM, ION-IMPLANTATION, GRAIN GROWTH, ERBIUM, RUTHERFORD BACK-SCATTERING, DIFFUSION CONSTANTS, INFRARED, SYNCHROTRON

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

The influence of high dose implants of erbium on the grain growth of titanium has been studied. Titanium samples implanted with erbium were annealed at 750°C. The grain structure of each sample was then studied using optical metallographic techniques. Data shows that erbium inhibits grain growth on both the implanted and surrounding regions. The inhibition of grain growth in the regions adjacent to the implanted region is due to surface segregation and diffusion of the erbium. Both (over)

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(continued)

the surface and bulk diffusion constants for erbium in titanium are estimated from Rutherford back-scattering analysis of the erbium profiles in titanium during heat treatments. The use of synchrotron radiation as a continuum infrared source has been investigated with respect to its feasibility in new national facilities under construction.

INHIBITION OF GRAIN GROWTH IN TITANIUM BY ION IMPLANTATION*

J. M. Cathcart, K. O. Legg, J. R. Stevenson and H. Solnick-Legg
School of Physics
Georgia Institute of Technology
Atlanta, Georgia

The influence of high dose implants of erbium on the grain growth of titanium has been studied. Titanium samples implanted with erbium were annealed at 750°C. The grain structure of each sample was then studied using optical metallographic techniques. Data is presented which shows that Er inhibits grain growth on both the implanted and surrounding regions. The inhibition of grain growth in the regions adjacent to the implanted region is due to surface segregation and diffusion of the Er.

*This work was supported by the Air Force Office of Scientific Research under Grant No. 79-0011.

I. Research Objectives

The long range objective of this research effort is a basic approach to the study of high dose ion implantation in titanium, aluminum, and alloys of Ti-Al. The study includes determination of the depth profile of the implanted species and the kinetics of this profile with temperature cycling and grain size changes as well as during oxide and nitride formation. The growth kinetics of oxide and nitride formation will be followed in ion implanted specimens to identify the important controlling mechanisms. Radiation damage associated with the implantation process will be evaluated in terms of its effect on growth kinetics and grain size distribution.

During the first year of the research effort the objectives included the development of instrumentation and techniques to achieve the long range objectives as well as a study of the effects of implanted species on grain size growth kinetics. Both platinum and erbium were chosen as ion implants.

II. Status of the Research Effort

Additional details concerning the status of the research effort can be found in the renewal proposal for work beginning November 1, 1979 as well as the material referenced in Section III of this report as submitted for publication. In brief the status of the research is as follows:

A) Grain Size Kinetics

The influence of high dose implants of erbium on the grain growth of titanium has been studied. Titanium samples implanted with erbium were annealed at 750°C. The grain structure of each sample was then studied using optical metallographic techniques. Data showed that Er inhibits grain growth on both the implanted and surrounding regions. The inhibition of grain growth in the area adjacent to the implanted region is due to surface segregation and diffusion of the Er.

While high temperature annealing produced large grain growth in pure titanium, erbium implanted titanium samples exhibited no grain growth at the

surface. This inhibition is not only in the implanted region but also on the whole surface surrounding the implant region. Calculations showed that erbium should segregate to the surface of titanium as is found experimentally. In addition, diffusion coefficients for erbium were estimated to be at least $5x10^{-5}$ cm^2 s^{-1} for the surface and approximately 10^{-15} cm^2 s^{-1} for the bulk.

Some preliminary work on platinum implants, reported in our renewal proposal, indicated that platinum also inhibited grain growth. Additional work on both types of implants is continuing.

The results on grain growth inhibition by implants of erbium lead us to believe that we can freeze the surface grain size distribution while studying the high temperature oxidation kinetics of titanium surfaces, thus insuring that the effects of grain boundary diffusion are held constant.

B) Techniques and Instrumentation

A variety of different techniques are available for the basic research studies on the high dose ion implantation specimens.

1) Specimen Preparation - Polycrystalline samples of pure titanium are cut from a half-inch diameter rod and annealed at 650°C for 45 minutes to eliminate acicular α and obtain an equiaxed grain structure. Following the pre-anneal the samples are mechanically ground and polished, electropolished and etched with Kroll's reagent. Grains are observed and photographed using standard metallurgical optical microscopy techniques.

2) Ion Implantation - A commercial ion implanter (a 200 M P R Implanter by Accelerators, Inc.) is used to create ion implants in the titanium surfaces. The facility within the School of Physics at Georgia Tech has been operational for several years and provides for implanting specimens under controlled environmental conditions in ultra-high vacuum with in situ surface diagnostics available.

3) Rutherford Backscattering - The current research uses 400 keV He^{++} ions as projectiles for studying the depth profile of the implanted species.

The change in profile with heat treatment permits estimates of both the bulk and surface diffusion constants.

4) Auger Spectroscopy - Auger spectroscopy is used in situ to determine the chemical composition of the surface both prior to implanting and after heat treatment, as well as to monitor oxidation states.

5) Oxidation Studies - A thermobalance for weight gain measurements has been moved to a convenient location in metallurgy and is currently being placed into operational condition. No studies using the thermobalance have been made during the first year.

6) Optical Instrumentation - Several studies have been completed by our research group to determine the optical constants of titanium and titanium exposed to oxygen. The studies have been from the vacuum ultraviolet through the visible portion of the electromagnetic spectrum. A new far infrared waveguide laser from Advanced Kinetics is now operational in our laboratory. Initial measurements will begin during the second contract year on using far infrared as a probe of the oxide formation on titanium surfaces.

7) Synchrotron Radiation as an Infrared Source - In looking at the techniques necessary in the near future for the study of titanium and other metals undergoing oxidation, a strong continuum in the infrared which is compatible with surface physics techniques will be required. Our research group has experience in the use of synchrotron radiation for optical studies in the vacuum ultraviolet and has published feasibility studies on the use of synchrotron radiation in the infrared. We are continuing computer calculations and have interacted with both the University of Wisconsin and Brookhaven National Laboratory relative to the establishment of an infrared port on new national facilities under construction.

III. Written Publications

The following publications have been published or submitted for publication.

1. "Optical Properties of Titanium and Titanium Oxide Surfaces," W. E. Wall, M. W. Ribarsky and James R. Stevenson, Journal of Applied Physics (to be published).

2. "Design Considerations for Parasitic Use of Synchrotron Radiation in the Infrared," James R. Stevenson and J. M. Cathcart, Proceedings of National Conference on Synchrotron Radiation Instrumentation, National Bureau of Standards (to be published).

3. "Inhibition of Grain Growth in Titanium by Ion Implantaion," J. M. Cathcart, K. O. Legg, J. R. Stevenson and H. Solnick-Legg, Proceedings of Material Research Society, November 1979 (to be published).

IV. Professional Personnel

The following personnel have been involved in the research.

Dr. James R. Stevenson - Professor and Principal Investigator
Dr. Keith O. Legg - Senior Research Scientist and Co-Principal Investigator
Dr. M. W. Ribarsky - Research Scientist and Co-Principal Investigator
Mr. J. M. Cathcart - Ph.D. Candidate
Mr. John M. Merboth - BS Candidate
Mr. Matthew J. Rutten - BS Candidate
Ms. Patricia L. White - BS Candidate
Ms. H. Solnick-Legg - Research Scientist (Metallurgist)

V. Interactions

A) Talks, Seminars, Poster Papers

1. "Design Considerations for Parasitic Use of Synchrotron Radiation in the Infrared," James R. Stevenson and J. M. Cathcart, National Conference on Synchrotron Radiation Instrumentation, National Bureau of Standards, Gaithersburg, Maryland, June 4-6, 1979.

2. "Synchrotron Radiation as an Infrared Source," James R. Stevenson, Seminar, Northern Illinois University, DeKalb, Illinois, Sept. 21, 1979.

3. "Synchrotron Radiation in the Infrared," James R. Stevenson and J. M. Cathcart, Eleventh Annual Synchrotron Radiation Users Group Conference, University of Wisconsin, October 23, 1979.

4. "Inhibition of Grain Growth in Titanium by Ion Implantation," J. M. Cathcart, K. O. Legg, J. R. Stevenson and H. Solnick-Legg, Materials Research Society, Cambridge, Mass., November 1979.

B) Personal Interactions

1. S. Fujishiso of Wright Patterson has been in both personal contact as well as written and verbal communication with our group relative to the implantation of platinum in turbine blade samples and tensile test specimens. Dr. Shiro Fujishiro is a member of the Structural Metals Branch of the Air Force Materials Laboratory at Wright Patterson Air Force Base.

2. T. Eckler of Pratt and Whitney Aircraft has conducted some fatigue tests and some oxidation studies on samples our group has implanted. The communication was established by Dr. Fujishiro.

3. Our group has communicated with both the University of Wisconsin National Synchrotron Radiation Facility and the Brookhaven National Sychrotron Facility at Meetings as well as by telephone.

4. Dr. Legg attended a conference in Washington on DOD research in the chemical sciences.

VI. No discoveries have been made or patent applications filed during the first year.